

THE BUSINES DEANORS (CA

TO ALL TO WHOM THESE; PRESENTS SHALL COME;

UNITED STATES DEPARTMENT OF COMMERCE

United States Patent and Trademark Office

REC'D 0 1 APR 2004

PCT

WIPO

February 12, 2004

THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A FILING DATE UNDER 35 USC 111.

APPLICATION NUMBER: 60/441,964

FILING DATE: January 23, 2003

PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN COMPLIANCE WITH RULE 17.1(a) OR (b)

By Authority of the

COMMISSIONER OF PATENTS AND TRADEMARKS

E. BORNETT

Certifying Officer

0124-03

PTO/SB/16 (10-01) Approved for use through 10/31/2002. OMB 065;-9032 U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control numbers U.S.

PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c)

EL 810961398 US Express Mail Label No.

	IM	VENTOR(5)				5
Given Name (first and middle [if any])	Family Name or Surname			Residence (City and either State or Foreign Country)			10/1 · -
Sumita Sanjeevi	Ranganathan			Ontario, Canada			- 37
Theodore John	Lang			Ontario, Canada			
Marek Andrei	Szymula ⁻			Ontario, C			
Additional inventors are being named on the separately numbered sheets attached hereto						·	
TITLE OF THE INVENTION (500 characters max)							
HIGH TEMPERATURE AND HIGH HUMIDITY RELEASE COATING FOR POLYMER FILM							.M
Direct all correspondence to:	CORRESPO	NDENCE A	DRESS				
Customer Number	26691		\rightarrow		Customer		l : f
OR Type Custo	omer Number here	-		Barc	ode Label i	LIGIÐ	!
Firm or Individual Name							
Address	,						
Address					<u> </u>		}
City		State	· · · · · · · · · · · · · · · · · · ·	· ZIP	<u> </u>		
Country		Telephone		Fax.	<u> </u>		
ENCLOSED APPLICATION PARTS (check all that apply)							 [
	Specification Number of Pages 17 CD(s), Number						
The state of the s	Other (specify)]
Application Data Sheet. See 37 CFR 1.76							
METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT Applicant claims small entity status. See 37 CFR 1,27. FILING FEE							
Applicant dains small entity status, see 57 CPR 1.27 A check or money order is enclosed to cover the filing fees AMOUNT (\$)							
The Commissioner is hereby authorized to charge filing fees or credit any overpayment to Deposit Account Number: Payment by credit card. Form PTO-2038 is attached.					· . .		
The Invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.							
✓ No. ✓ Yes, the name of the U.S. Government egency and the Government contract number are:							
Respectfully submitted,		<u>.</u>	Date	01/23/2003] _		
SIGNATURE REGISTRATION NO. 50,157					·		
TYPED or PRINTED NAME Thomas R. Mancini (if appropriate) Docket Number: 2609				26090-0	25		
TELEPHONE (302) 984-6127 26090-025							

USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT

This collection of Information is required by 37 CFR 1.51. The information is used by the public to file (and by the PTO to process) a provisional application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 8 hours to complete, including gathering, preparing, and submitting the complete provisional application to the PTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, Washington, D.C. 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Box Provisional Application, Assistant Commissioner for Patents, Washington, D.C. 20231.

Please type a plus sign (+) inside this box

PTC/SB/21(08-00)
Approved for usé through 10/31/2002. OMB 0851-0031
U.S. Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid GMB control number.

Under the P	eperwork Reduction Act of	f 1995, no persor	is are requir	ed to respond to a collection o	information unles	s it displays a valid CMB control nur	nber.	
TRANSMITTAL FORM (to be used for all correspondence after initial filing)		Application Number		Unknown	Unknown			
		Filing Date		Filed contemporaneously herewith				
		First Named Inventor		Sumita Sanjeevi Ranganathan		7 · · ·		
		Group Art Unit		Unknown	•			
		Examiner Name		Unknown				
Total Number of Pages i	tal Number of Pages in This Submission 22		Attorney Docket Number		26090-02	5.		
ENCLOSURES (check all that apply)							_	
		ment Papers Application)		After Allowance Communication to Group				
Fee Attached	Fee Attached Drawin		ı g(s) .		Appeal Communication to Board of Appeals and Interferences			
Amendment / Response ' Licens		ing-relate	d Papers		Communication to Group Notice, Brief, Reply Brief)			
. After Final : Affidavits/declaration(s)			n		Proprie	Proprietary Information		
Extension of Time	Request	Petition to Convert to a Provisional Application			· 🔲 Status	Letter		
Express Abandon	ment Request	Power of Attorney, Revocation Change of Correspondence Addres			Other identify	Enclosure(s) (please y below):		
Information Disclo	sure Statement	☐ Terminal Disclaimer		Cover	sional Patent Application Sheet lication			
Certified Copy of I	Priority	Request for Refund						
Response to Missing Parts/ Incomplete Application			umber of	CD(s)				
Response to Missing Parts under 37 CFR 1.52 or 1.53								
SIGNATURE OF APPLICANT, ATTORNEY, OR AGENT								
Firm or Thomas R. Mancini Registration No.: 50,157 Individual name :								
Signature	Signature / / / / / / / / / / / / / / / / / / /							
Date January 23; 2003								
CERTIFICATE OF MAILING						$\overline{}$		
I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Assistant Commissioner for Patents, Washington, D.C. 20231 on this date January 23, 2003						pe		
Typed or printed name Darcy A. White								
Signature Date January 23, 2003								

Burden Hour Statement: This form is estimated to Jake 0.2 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form snould be send to the Chief Information Officer, U.S. Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

TITLE .

Improved High Temperature and High Humidity Release Coating for Polymer Film

FIELD OF THE INVENTION

[0001] The present invention relates to an improved release coating composition that may be applied to a film that may then be used as a substrate useful for applications requiring release for a broad range of temperatures and high humidity conditions, which temperatures may range from about 20°C to about 210°C. These applications include release substrate used in the manufacture of calendared cured sheet rubber and molding paste composites, such as sheet molding compound (SMC), thick molding compound (TMC), bulk molding compound (BMC) and fiberglass composites,

BACKGROUND OF THE INVENTION.

[0002] In the rubber industry, sheets of cured rubber compound are prepared by a calendaring process. Typically these sheets are from about 100 to about 400 feet in length. The uncured rubber sheet is laid onto a supporting interleaf film or sheet and then the two sheets are wound onto a mandrel. The interleaf is usually cellophane or silicon coated paper. The interleaf does not melt at the curing temperature and prevents the sheets from fusing with each other during the curing process. Sometimes talc or zinc stearate is applied to the interleaf to enhance release of rubber sheets from the interleaf after curing. Subsequently, the roll of rubber and interleaf can be over wound and held under tension using an over-wrap, which can be any film or cloth having good tensile properties that tends to shrink at oven curing temperatures. The cured sheet rubber may be used as components for aircraft engines and gaskets for rubber roofing membranes. Teflon[®] sheets, talc dust, and cloth are commonly used as interleaves in the rubber industry.

[0003] SMC is a composite material and usually comprises crosslinkable polymeric resin, most often unsaturated polyester resin; styrene monomer, plus catalyst; particulate filler, such as calcium carbonate; chopped glass

fiber reinforcement; and various other additives in minor amounts, such as pigments and other modifiers.

[0004] The manufacture of SMC begins by laying the paste combrising all ingredients except the glass fibers, on a bottom carrier or release sheet, i.e., a film. The glass fibers are poured on top of the resin. More paste is poured over the glass fibers. A top carrier release sheet is laid down, and the edges of the top and bottom sheets are folded over to form a sandwich. The film and hence the composite is then kneaded to mix the glass fibers and the paste. The sandwich is then festooned (folded back and forth in a continuous fashion) into a bin and stored for up to about 14 days to cure or mature. Satisfactory results may be obtainable after as little as 2.5 days, but often more time is required. During this time the viscosity of the composite increases significantly (approximately ten-fold). [0005] At the end of the curing period, the carrier release films, top and bottom are stripped away, the solidified SMC is cut and put into a heated press. In roughly one minute or less, out comes a semi-finished product, such as an auto part, for example, an automobile hood. [0006] TMC is produced by a different machine and a process different from those used for producing SMC. Although TMC is prepared as a continuous length of material, it is cut into slabs for curing and storage

because it is thicker than SMC. SMC is usually 1" thick, but may range from ½" to 3" in thickness. TMC may range from ½" to 4" in thickness. TMC is stronger because some of its fiberglass fibers may be positioned vertically, and more filler may be added. A most significant difference between SMC and TMC is that in making TMC, the glass fibers are mixed with the paste prior to being deposited on the carrier or release film, and thus no kneading of the composite sandwich is necessary when TMC is made into slabs. This therefore places different requirements on the carrier or release film as tear strength may not be as critical for carrier release film used to make TMC.

[0007] BMC is also a composite material of resins, fillers and reinforcements. Typically, it comprises 30% resins, 50% fillers and additives and 20% reinforcement, such as glass fiber. It may also contain

catalysts. The high filler loadings can provide improved stiffness and fire retardence. BMC is manufactured by preparing a putty-like molding compound comprising the above-noted components in a "ready to mold" form. Molding pressures usually range from about 350 to 2000 psi at temperatures of between 121 and 176°C. BMC can be made into precise shapes with various types of inserts, and therefore the moldings can be extremely complex. One limitation of BMC is the loss of strength caused by degradation of glass fiber reinforcements during energy-intensive mixing.

[0008] BMC is primarily used as a replacement for cast metals. The actual physical characteristics of BMC are determined primarily by the choice of resin and desired end use. Possible end uses include electrical grade; low shrink/general purpose; appliance/structural; low profile; automotive grade; and corrosion resistant. Major applications of BMC include air conditioner components; pump housings; circuit breakers; computer and business equipment components; garbage disposal housings; motor parts; power tools; gear cases; electrical insulators; and circuit covers. [0009] In selecting a carrier release film there are some basic requirements or properties that are preferably met for the film to be suitable. While styrene barrier, moisture barrier, and mechanical strength are relevant, most important are release from the paste composite, be it SMC, BMC, or TMC, and the cost of the release film. [0010] Nylon films represent a potential replacement for silicon-coated paper and cellophane as interleaves in the rubber calendaring industry, because of their high tensile strength. The plain Nylon films work quite well as release films in the case of some but not all rubber samples. Apart from sticking to the sheets of rubber, the nylon film sometimes causes wrinkles on the surface of the cured rubber. It is speculated that gases emanated during curing of rubber cause such wrinkles. [0011] Cellulose ethers are water-soluble polymers derived from cellulose. A commercially available cellulose ether is available under the Methocel® brand from The Dow Chemical Company. These products are available in various viscosity grades, ranging from 3 to over 200,000 mPa's.

Generally, these viscosities refer to the viscosity of a 2% Methocel® solution in water at 25°C. The methylcelfulose products include hydroxypropyl substituted cellulose ethers. Such products are also available from other sources such as China Yixing Kaili Chemical Pharmaceutical Factory of Yixing city, Jiangsu, China; Carbomer Inc of Westborough, MA; and Penta Mnfg. Co. of Livingston, NJ. Methocel® products are used as mold-release agents, stabilizers, and thickeners in rubber latexes, where they contribute also to more uniform drying and less pinholing (see Dow METHOCEL® Cellulose Ethers Technical Handbook available from The Dow Chemical Company Website, July 2000).

BACKGROUND ART

there is disclosed a coating composition for use as a surface coating for polymer release films for use in high temperature and/or high humidity applications, which comprises a solution of at least one hydroxypropyl methylcellulose having hydroxypropyl molar substitution of from 0 to about 0.82.

[0013] Various attempts have been made to make and coat non-stick coatings to film or film structures used for high temperature applications. Some of the patents pertaining to release coatings include U.S. Pat. No. 5,139,835 to Kitamura et al.; U.S. Pat. No. 3,503,773 to Bisschops et al.; U.S. Pat. No. 4,956,233 to Chu et al; U.S. Pat. No. 4,956,241 to Chu et al.; U.S. Pat. No. 3,945,404 to Yamamatsu et al.; U.S. Pat. No. 5,547,738 to Mitchell et al.; U.S. Pat. No. 5,492,599; U.S. Pat. No. 2,273,040; U.S. Pat. No. 3,484,271 to Kaliski et al.; Japanese Examined Patent Application 63,075,199 (Kanzaki Paper); U.S. Pat. No. 4,226,749; U.S. Pat. No. 3,976,490; U.S. Pat. No. 5,959,031 issued to Thurgood; U.S. Pat. No. 3,837,375 to Higgins et al; European Patent EP 0295375A2; U.S. Pat. No. 5,858,487 to Boehler et al; and U.S. Pat. No. 4,735,860. [0014] The various types of release materials can be categorized as waxes, such as petroleum waxes, vegetable waxes, animal waxes, and synthetic waxes; fatty acid metal soaps, such as metal stearates and others, for example, calcium ricinoleate; other long chain alkyl derivatives,

fatty esters, fatty amides and amines, fatty acids and alcohols; polymers, such as polyolefins, silicones, fluoropolymers, natural polymers; others like poly(vinyl alcohol) and polyoxyalkylenes; fluorinated compounds and fluorinated fatty acids; and inorganic materials, such as silicates, talc, clays, kaolin, mica, and other particulates such as silica, graphite and carbon.

[0015] While all of the above references propose release coatings of various types, there remains a need for effective, inexpensive, high temperature, high humidity release coatings which can be applied to thermally resistant polymer films and which do not permanently transfer off the film to the surface in contact therewith.

[0016] Traditional release agents such as erucamide and polytetrafluoroethylene, which bloom to the surface in polyolefins, fail to do so in case of nylon films, such as polyamide66. Apparently, polyamide66 films have higher surface tension (43-50dyn/cm), can absorb up to 2% by weight of water and can be heated up to 150°C with no degradation. All these properties make polyamide66 film a friendly substrate for coating with water based coatings.

[0017] The disclosures of all documents, patents and applications referred to herein are incorporated herein by reference.

SUMMARY OF THE INVENTION

[0018] Thus in one aspect, the present invention provides a coating composition for use as a surface coating for polymer release films for use in at least one of high temperature and high humidity applications, which comprises a solution of at least one hydroxypropyl methylcellulose having hydroxypropyl molar substitution of from 0 to about 0.82 in combination with at least one water-borne fluorochemical additive selected from perfluoralkyl methacrylic acid copolymers.

[0019] In another aspect, the invention provides a composition as defined above, wherein the amount of the at least one hydroxypropyl methylcellulose having hydroxypropyl molar substitution of from 0 to about 0.82, preferably 0.21 and comprises from about 27% to about 50% by weight of solids in the solution, while the amount of the fluorochemical

additive comprises from about 73% to about 50% by weight of solids in the solution. The solution includes water and an organic solvent, alcohol being an example of the latter. The amount of water in the solution may range from about 80% to about 10% by weight of total solution. The amount of organic solvent may range from about 20% to about 90% by weight. The percentage of solids in solution is derived from the methylcellulose and the perfluoroalkyl methacrylic copolymer. In the case of the methylcellulose component, this is about 100% by weight solids, while the fluoro additive typically comprises about 30% by weight solids. Generally, the percent by weight solids in the solution is kept very low, the range being from about 5% to about 1% by weight. In the case of a typical composition, the amount of solids range from about 1% to about 2% by weight.

[0020] In another aspect, the invention provides a process for coating the surface of a polymer film to provide a release film for use in high temperature and/or high humidity conditions, which comprises coating at least one surface of the polymer film with a solution as described above to provide a coating weight of at least about 0.004 lb/ream per side and drying the coated film to set the coating. Generally, the range is from about 0.1 lb/ream per side to about 0.3 lb/ream per side. In another embodiment of this process, the film is coated on both sides in separate passes or in a single pass to achieve the desired coating weight.

[0021] In yet another aspect, the invention provides a release polymer film coated on at least one surface with a solution as described above.

[0022] The release coating of the present invention has been found to be useful in cured rubber manufacturing applications and also has utility in the manufacture of SMC, BMC and TMC, as well as fiberglass composites. In addition, it is useful in applications such as those described in US Patents Nos. 3,837,375 (packaging of hot, highly viscous, tacky polymers such as low molecular weight polystyrene); 5,858,487 (laminated, non-stick food wraps); and 4,735,860 (therma-sensitive transfer sheets); as well as EP 0 295 375 (cyanate resin-based prepregs and films for use in advanced structural materials).

[0023] In another aspect the invention provides a process for curing rubber which comprises forming a sheet rubber layer in a calendar, laying layers of a release film as described above between layers of the sheet rubber, tightly overwrapping the stack of layers with a release film or cloth, before subjecting the stack of layers to elevated temperature in a dry or steam oven wherein the sheet rubber or sheet molding compound is cured and subsequently unwrapping the stacked, cured sheets.

[0024] In another aspect the invention provides a process for producing

(a) casting a layer of heat-curable thermosetting resin, in fluid form, onto a continuously advancing polymeric release film;

sheet molding composites which comprises:

- (b) introducing reinforcing material onto the advancing fluid layer;
- (c) laying a polymeric film on the top surface of said reinforced fluid layer thereby forming a sandwich composite;
- (d) advancing said sandwich composite through a series of kneading and compaction rolls; and
- (e) winding the sandwiched composite into a roll for partial curing; the improvement comprising using a release film as defined above. [0025] In another aspect the invention provides a process for making thick molding composites, comprising
 - (a) introducing reinforcing material into a heat-curable thermosetting resin, in fluid form and mixing same until the material is mixed and wetted:
 - (b) casting a layer of said mixture onto a continuously advancing polymeric film:
 - (c) laying a polymeric film on the top surface of said reinforcing material-resin layer to form a sandwich composite;
 - (d) advancing the sandwich composite through at least one compaction roll;
 - (è) cutting the continuous lengths of the sandwich composite into lengths for partial curing;

the improvement comprising using a release film as defined above.

[0026] There are other fluoro based aqueous release coating compositions available commercially. Typically, these formulations are dispersions, while the improved formulation of this invention is a uniform solution, which is more amenable to coating using a gravure process.

DETAILED DESCRIPTION OF THE INVENTION

[0027] In the following Table 1 there is set out the hydroxypropyl molar substitution of various grades of hydroxypropyl methylcellulose available commercially from the Dow Chemical Company, which have been found to be useful in the present invention.

TABLE 1 DIFFERENT GRADES OF HYDROXYPROPYL METHYL CELLULOSE				
Product	Hydroxypropyl molar			
	substitution			
Methocel [®] A	0			
Methocel [®] E	0.23			
Methocel [®] F	0.13			
Methocel [®] J	0.82			
Methocel [®] K	0.21			

[0028] Methocel A does not contain any hydroxypropyl groups.

Methocel(s) E, F, J and K contain varying ratios of hydroxypropyl and methyl substitution, a factor that influences organic solubility and thermal gellation in aqueous solutions.

[0029] Hydroxypropyl methyl cellulose grades can be classified into high viscosity and low viscosity grades. Low viscosity grades of hydroxypropyl methyl cellulose are those grades, which at solution concentration of about 2% in water at room temperature (20°C) result in the solution viscosity in the range of 0.1-100 centipoise. A viscosity of 35 centipoise seems to be optimum, a lower viscosity (3 centipoise) leads to transfer. High viscosity grades of hydroxypropyl methyl cellulose are those grades, which at

solution concentration of about 2% in water at room temperature (20°C) result in the solution viscosity in the range of 100-100,000 centipoise. The solutions of surface treated grades of hydroxypropyl methylcellulose available from Dow Chemical Company in water require pH adjustment in order to trigger the hydration process and subsequently, the viscosity build up. The latter trigger can be conveniently achieved by adding a small amount of base, such as ammonium hydroxide to the dispersion of surface treated hydroxypropyl methylcellulose in water.

[0030] The type of fluorochemical additive found to be effective in the improved formulation of the invention is selected from fluorochemical additives that are excellent wetting and leveling agents. They reduce the surface tension of water and are capable of creating surface tensions as low as 18 dyn/cm. A commercial example of this type of fluorochemical additive is water soluble perfluoroalkyl methacrylic acid copolymers available from E.I. du Pont de Nemours under the Trademark ZONYL. Specific water-borne fluorochemical additives are ZONYL 8740, 9338, 9361, 8867L and 9360, as well as ZONYL NWG.

[0031] The range for the amounts of the methylcellulose and the fluorochemical additive which may typically be added to the formulation are as follows:

Range:

lower limit: 0.4% methylcellulose solid + (3.5% fluorochemical = 1.05% solids) = 28% methylcellulose and 72% fluorochemical in the solids upper limit: 0.9% methylcellulose solid + (3.0% fluorochemical = 0.9%) = 50% methylcellulose and 50% fluorochemical in the solids

Defining this range in terms of solids:

27-50% methylcellulose, >50% results in transfer to the substrate, and about 25% is required to bond the fluorochemical onto the Nylon film, we did attempt coating the fluorochemical alone and were not successful. A minimum amount of methylcellulose as binder is required to get the fluorosurfactant fluorochemical onto the Nylon film.

73-50% fluorosurfactant fluorochemical: This additive is responsible for the release characteristics at very very low levels. It would work quite well

by itself if it could be coated onto the film, it does not bond to the Nylon film and so needs a binder.

BRIEF DESCRIPTION OF THE DRAWINGS

[0032] In the accompanying drawings which are used to illustrate the present invention only,

Figure 1 is a photograph of silicone sheet which shows patches of coating remaining after a prior art release film has been removed;

Figure 2 is a photograph of a silicone sheet which shows scattered spots of coating remaining after removal of a prior art release film and after talcing:

Figure 3 is a photograph of silicone sheet which shows spots of coating remaining but only on talcing;

Figure 4 is a photograph of silicone sheet which shows very few spots of coating remaining but only on talcing;

Figure 5 is a photograph of silicone sheet which shows no transfer at all and to which has been applied a release film of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

EXAMPLE

[0033] Composition:

Isopropyl alcohol: 15%

Methocel K35LV (Hydroxypropyl methylcellulose): 0.4 to 0.9%

Zonyl 8740 (Perfluoroalkyl methacrylic copolymer, 30 wt% in water): 3.5

to 3.0%

Water: 81.1%

[0034] Coating preparation procedure:

Added the appropriate amount of Methocel K35LV slowly with constant stirring to the appropriate amount of Isopropyl alcohol. Stirred for ½ hour until a homogenous dispersion is formed. Added water to the above dispersion with constant stirring. The preweighed amount of Zonyl was added to the above solution with stirring. Coating Procedure: gravure coated onto Nylon 66 (Dartek) film using a 180 quad cylinder. Coat Weights: about 0.06 lbs/ream/side.

[0035] Examples of Invention:

- Nylon 66 film coated with the new formulation was tested on several rubber samples for the following performance criteria:
- .1. Release: ease with which the film sample can be peeled off the rubber after curing.
- 2. Transfer: extent to which the coating material transfers onto the cured rubber surface, leading to marking that becomes more prominent on applying talc. Measured by visual inspection. Mostly an aesthetic issue but in some cases where the rubber sheet is double plied, transfer leads to reduced adhesion between the sheets.

[0036] Comparisons:

Uncoated Nylon 66 film:

- does not show adequate release from Silicone rubbers (several colours from several customers were tested: brown, orange, red, grey and tan.

 The colour chemistry seems to affect the performance of the films.)
- does not show adequate release from Neoprene rubbers (white and black).
- does not release from viton (black)
- does not release from most grades of Nitrile rubbers (black and white).
- does not release from Butyl rubbers (natural and black).
- does release from very few grades of neoprene(black), epdm, silicone rubbers.

[0037] Coated product of prior art (U.S. Application Serial No. 09/909,746):

- shows good release from Silicone rubbers (brown, red, grey), Neoprene, Viton, Nitrile and Butyl rubbers.
- does not release from tan and orange Silicone.
- extensive transfer of the coating onto the rubber in the case of Silicones (brown and red), Viton (black), Nitrile (Black and White), Neoprene (black), EPDM (black).
- extensive transfer in the case of white Nitrile sample hindering adhesion in double plying.

[0038] Coated product of invention:

-good release and minimum transfer at levels acceptable by visual inspection in all the above cases from the following rubber samples Silicones (brown, red, orange, tan, grey)

Vitons (black)

Nitriles (black and white)

Neoprene (black)

EPDM (black).

:Butyl (natural and black)

No adhesion issues in double plying in the case of white Nitrile. [0039] The coating of the composition can be performed by rolling, dipping or spraying. The rolling method is preferred. Details of these coating methods are well known to those skilled in the art. Drying of the coated film is preferably by air drying, in a heated oven, at a temperature in the range of about 40°C to about 120°C.

[0040] The humidity conditions under which the release film performs range from 0 to about 100% relative humidity. The high temperature conditions range from about room temperature (20°C) to about 210°C. [0041] The solution of methylcellulose may comprise a binary mixture of an organic solvent and water. There are a variety of organic solvents that may be used in such a binary mixture and the organic solvent may be selected from glycols, esters and amines. The Dow Technical Handbook for Methocel Cellulose Ethers referenced earlier contains a listing of suitable specific solvents. The solution may be prepared in concentrated form and then diluted to an appropriate concentration for the desired coating weight.

[0042] The polymer film may be selected from polyolefins, polyesters, nylons and blends thereof. Nylon 66, Nylon 6 and polyester films are preferred. The films may be monoaxially or biaxially oriented. Generally any film having a softening point above the temperature of the application for the coating may be used. A preferred film is monoaxially oriented (in the machine direction) nylon, in particular nylon 66. A commercial example is Dartek T404 available from Enhance Packaging Technologies Inc. This film has good MD shrink properties at rubber curing conditions.

[0043] In the following examples of the invention, a 0.001" thick. monoaxially oriented, annealed at 150°C, polyamide66 film supplied by Enhance Packaging Technologies Inc. of Whitby, Ontario under the trade name Dartek® T404 was coated using a direct gravure coater supplied by - Faustel Inc. of Germantown, Wisconsin. DuPont Dow Elastomers Inc. of Akron, Ohio supplied the uncured rubber compound used for dry and wet. release testing, under the commercial name of Viton®. The latter rubber compound contained 100 parts of Viton® fluoroelastomer A201C, 3 parts of MgO, 6 parts of Ca(OH)2 and 30 parts of carbon black. [0044] In the Examples, the coat weight of coated release film was determined by washing off the coating with water from the 8"x 8" square sample. The weight loss in grams was multiplied by a factor of 14.9 to obtain the coat weight in lb/ream or in g/0.61m². The latter technique was recommended by Morton International, Inc. Packaging Adhesives North America of Woodstock, Illinois in document #W-3020-641-02 dated May 15, 1994.

RELEASE TESTING PROTOCOL

[0045] Two types of curing equipment are commonly used for curing rubber in the rubber release industry, viz. steam and gas ovens.

[0046] In a dry release test, approximately 2.5g of rubber are pressed in between two release sheets at 10,000 pounds pressure in a 9inch x 9 inch (22.86cm x22.86cm) press and 350°F (176°C) for 1hr (most silicone rubbers are for 10 minutes). The time required here is rubber dependent. At least 35 minutes is required for Viton® rubber. Subsequently, the sample is cooled to room temperature and a strip (4"x1/2") of the release sheet / rubber / release sheet sandwich is cut and subjected to a 90 degree peel test at a crosshead speed of 10"/min using a peel tester. The peel test is conducted as per the Pressure Sensitive Tape Council's appendix B and ASTMD1876. Curing conditions are rubber sample dependent. The dry release test is designed on the assumption that humidity has no effect on peel strength.

[0047] A wet release test is designed such that the high humidity curing condition is taken into account. As per this test, a 6"x3" sheet of uncured

Viton® rubber compound interleaved with the release film is rolled onto a stainless steel core, 6" long having an outer diameter of ¾". A cross-sectional view of the rubber interleaved with the release film mounted on the core is shown in Figure 6(A). The latter roll is over-wrapped with a masking tape. This roll is fed into an autoclave for curing at 160°C, 80psi steam The rubber roll is kept in the autoclave under latter conditions for 6 hours before cooling down the autoclave and taking out the cured rubber rolls. On cooling the steam condenses and collects at the bottom of the clave. The rolled rubber samples are placed on a wire mesh to prevent them from being submerged in the water. Subsequently, the rubber rolls are unwrapped and the force required to peel off the release film from the cured rubber is determined as per the Pressure Sensitive Tape Council's appendage B and ASTMD1876.

WETTABILITY OF DARTEK®T404

[0048] Zonyl by itself does not wet the Dartek T404 film, but the combination of the invention does.

[0049] The invention may be varied in any number of ways as would be apparent to a person skilled in the art and all obvious equivalents and the like are meant to fall within the scope of this description and claims. The description is meant to serve as a guide to interpret the claims and not to limit them unnecessarily.

WE CLAIM:

- 1. A coating composition for use as a surface coating for polymer release films for use in at least one of high temperature and high humidity applications, which comprises a solution of at least one hydroxypropyl methylcellulose having hydroxypropyl molar substitution of from 0 to about 0.82 in combination with at least one water-borne fluorochemical additive selected from perfluoralkyl methacrylic acid copolymers.
- 2. A composition as claimed in Claim 1, wherein the amount of the at least one hydroxypropyl methylcellulose having hydroxypropyl molar substitution of from 0 to about 0.82 comprises from about 27% to about 50% by weight of the solids in the solution, while the amount of the fluorochemical additive comprises from about 73% to about 50% by weight of solids in the solution.
- 3. A composition as claimed in Claim 1, wherein the solution includes water and an organic solvent.
- 4. A composition as claimed in Claim 1, wherein the solution comprises alcohol and water and the amount of water in the solution may range from about 80% to about 10% by weight of total solution and the amount of organic solvent may range from about 20% to about 90% by weight.
- 5. A composition as claimed in Claim 1, wherein the percent by weight solids in the solution is less than about 2% by weight.
- 6. A process for coating the surface of a polymer film to provide a release film for use in high temperature and/or high humidity conditions, which comprises coating at least one surface of the polymer film with a solution as claimed in Claim 1 to provide a coating weight of from at least about 0.004 lb/ream to about 0.3 lb/ream per side and drying the coated film to set the coating. In another embodiment of this process, the film is coated on both sides in separate passes or in a single pass to achieve the desired coating weight.
- 7. A process as claimed in Claim 6, wherein the coating weight is from about 0.1 lb/ream per side to about 0.3 lb/ream per side.
- 8. A process as claimed in Claim 6, wherein the release polymer film is coated on at least one surface.

- 9. A process for curing rubber which comprises forming a sheet rubber layer in a calendar, laying layers of a release film obtained by the process of Claim 6 between layers of the sheet rubber, tightly overwrapping the stack of layers with a release film or cloth, before subjecting the stack of layers to elevated temperature in a dry or steam oven wherein the sheet rubber or sheet molding compound is cured and subsequently unwrapping the stacked, cured sheets.
- 10. A process for producing sheet molding composites which comprises:
 - (a) casting a layer of heat-curable thermosetting resin, in fluid form, onto a continuously advancing polymeric release film;
 - (b) introducing reinforcing material onto the advancing fluid layer;
 - (c) laying a polymeric film on the top surface of said reinforced fluid layer thereby forming a sandwich composite;
 - (d) advancing said sandwich composite through a series of kneading and compaction rolls; and
- (e) winding the sandwiched composite into a roll for partial curing; the improvement comprising using a release film obtained by the process of Claim 6.
- 11. A process for making thick molding composites, comprising
 - (a) introducing reinforcing material into a heat-curable thermosetting resin, in fluid form and mixing same until the material is mixed and wetted;
 - (b) casting a layer of said mixture onto a continuously advancing polymeric film;
 - (c) laying a polymeric film on the top surface of said reinforcing material-resin layer to form a sandwich composite;
 - (d) advancing the sandwich composite through at least one compaction roll;
 - (e) cutting the continuous lengths of the sandwich composite into lengths for partial curing;

the improvement comprising using a release film obtained by the process of Claim 6.

Improved High Temperature and High Humidity Release Coating for Polymer Film

ABSTRACT

A coating composition for use as a surface coating for polymer release films for use in at least one of high temperature and high humidity applications, which comprises a solution of at least one hydroxypropyl methylcellulose having hydroxypropyl molar substitution of from 0 to about 0.82 in combination with at least one water-borne fluorochemical additive selected from perfluoralkyl methacrylic acid copolymers.

Figure 1

TRANSFER RATING= 4, Patches NOT acceptable



Figure 2

TRANSFER RATING= 3, Scattered spots before and after talking, NOT acceptable

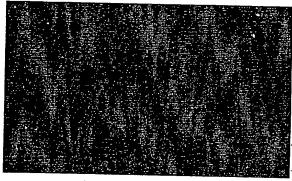


Figure 3

TRANSFER RATING= 2, Spots only on Talcing, not acceptable, NOT acceptable

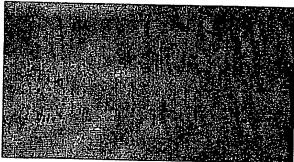


Figure 4



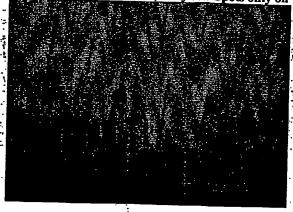


Figure 5

TRANSFER RATING=0, No Transfer at all

